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14. ABSTRACT Aluminum powders have long been used as additives in propellants, pyrotechnics and explosives. Aluminum has a high enthalpy of combustion but relatively low burn rate. Addition of reactive nanocomposite powders can increase the burn rate of aluminum and thus the overall reaction rate of the energetic formulation. Replacing only a small fraction of the fuel by a nanocomposite material can enhance the reaction rate with little change to the thermodynamic performance of the formulation. This research showed the feasibility of the above concept using nanocomposite powders prepared by Arrested Reactive Milling (ARM), a scalable “top-down” technique for manufacturing reactive nanomaterials. The nanocomposite materials used in this study were 2B+Ti, and Al-rich thermites: 8Al+3CuO, and 8Al+MoO ₃ . The reactive nanocomposite powders were added to micron-sized aluminum powder and the mixture was aerosolized and burned in a constant volume chamber. The combustion atmosphere was varied using oxygen, nitrogen, and methane. The resulting pressure traces were recorded and processed to compare different types and amounts of modifiers. Additives of nanocomposite powders of 8Al+MoO ₃ and 2B+Ti to micron-sized aluminum were found to be effective in increasing both the rate of pressure rise and maximum pressure in the respective constant volume explosion experiments. It was observed that 20 wt % of additive resulted in the best combination of the achieved burn rate and pressure.		
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Aluminum Burn Rate Modifiers Based on Reactive Nanocomposite Powders

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Abstract

Aluminum powders have long been used as additives in propellants, pyrotechnics and explosives. Aluminum has a high enthalpy of combustion but relatively low burn rate. Addition of reactive nanocomposite powders can increase the burn rate of aluminum and thus the overall reaction rate of the energetic formulation. Replacing only a small fraction of the fuel by a nanocomposite material can enhance the reaction rate with little change to the thermodynamic performance of the formulation. This research showed the feasibility of the above concept using nanocomposite powders prepared by Arrested Reactive Milling (ARM), a scalable “top-down” technique for manufacturing reactive nano-materials. The nanocomposite materials used in this study were 2B+Ti, and Al-rich thermites: 8Al+3CuO, and 8Al+MoO₃. The reactive nanocomposite powders were added to micron-sized aluminum powder and the mixture was aerosolized and burned in a constant volume chamber. The combustion atmosphere was varied using oxygen, nitrogen, and methane. The resulting pressure traces were recorded and processed to compare different types and amounts of modifiers. Additives of nanocomposite powders of 8Al+MoO₃ and 2B+Ti to micron-sized aluminum were found to be effective in increasing both the rate of pressure rise and maximum pressure in the respective constant volume explosion experiments. It was observed that 20 wt % of additive resulted in the best combination of the achieved burn rate and pressure.

Keywords: aluminum burn rate modifier, reactive nanocomposite, metal combustion

1 Introduction

Various nanocomposite materials are currently under development as potential components of different energetic formulations, from propellants to explosives, to pyrotechnics, e.g., [1 – 14]. The advantages anticipated from such materials are primarily due to a very developed reactive surface that facilitates rapid initiation of the exothermic reaction and results in a nearly adiabatic

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reaction temperature. At the same time, the overall energy outputs from many exothermic reactions employed in such materials, including thermites, intermetallic, and metal-metalloid compositions, are smaller than the benchmark values for aluminum combustion in air or in other practically important oxidizers (e.g., ammonium perchlorate). Thus, replacement of aluminum used as a fuel in most metallized energetic formulations with almost any of the nanocomposite materials currently under development would result in an overall reduction of the theoretical reaction enthalpy. This negative effect may be offset by an increase in the efficiency of metal combustion, so that the overall increase in practical performance is still anticipated. Therefore, the optimized composition would combine the high energetic output with the accelerated reaction rate. The approach discussed in this paper suggests that replacing only a fraction of aluminum fuel with a reactive nanocomposite material could result in an acceleration of the ignition kinetics for all metal fuel. Aluminum particles located in vicinity of the igniting reactive nanocomposite particles would be heated more efficiently and ignite sooner. It is anticipated that a relatively small addition of the reactive nanocomposite material would provide a number of localized hot spots within the igniting energetic formulation, which would accelerate ignition of the nearby aluminum particles, which, in turn, will accelerate ignition of their own particles-neighbors. Effectively, the nanocomposite material will serve as a burn rate modifier for an aluminized energetic formulation. The amount of such modifier is expected to be a function of the specific formulation. In this paper, the proposed concept is initially explored for aluminum particles burning in a gaseous oxidizer in presence of products of hydrocarbon combustion. Such environments are relevant for both enhanced blast explosives and metallized solid propellants.

2 Materials

Reactive nanocomposite powders were prepared by arrested reactive milling (ARM), a high-energy mechanical milling technique [8 – 14]. Samples of three micron-sized, fully dense nanocomposite powders with bulk compositions $2\text{B}+\text{Ti}$, $8\text{Al}+3\text{CuO}$, and $8\text{Al}+\text{MoO}_3$ were produced using a Retsch 400 PM planetary mill. Further details on the material synthesis are available elsewhere [13, 14]. Typically, the nanocomposite materials consist of micron-sized particles whereas each particle is a fully-dense, three-dimensional composite with characteristic dimension of material mixing of about 100 nm. Commonly, the morphology of composite is that of inclusions of one component, e.g., B, CuO, or MoO_3 , embedded into a matrix of another component such as Ti or Al. In this study, the nanocomposite materials prepared by ARM were added to a spherical aluminum powder, 10 – 14 μm nominal particle size by Alfa Aesar. The mixing of aluminum and nanocomposite powders was performed using a SPEX Certiprep 8000 shaker mill operated without milling balls for three minutes. The particle size distributions for all powders used in this project were measured using a Coulter LS 230 Enhanced Laser Diffraction particle size analyzer. The size distributions and respective volume mean particle sizes for all powders are shown in Figure 1.

3 Experimental

A common concern for all reactive metal powder additives is their sensitivity to electro-static discharge (ESD) ignition. All the materials used in this project were tested using a firing test system model 931 by Electro-tech Systems, Inc., according to Mil-1751A standard.

Constant Volume Explosion (CVE) experiments were performed with a set of materials including aluminum and aluminum mixed with different amounts of added nanocomposite burn rate modifiers. The details of the CVE experimental methodology and setup are described elsewhere [15 – 17]. Figure 2 shows a simplified diagram of the CVE apparatus.

The powders were introduced into a nearly spherical 9.2 liter vessel as an aerosol and ignited at the center of the vessel. The powder load was selected considering results of thermodynamic equilibrium calculations for combustion of aluminum in different gas mixtures used in experiments. It was predicted that the maximum adiabatic flame temperature for the 9.2 liter vessel filled with air occurs at an aluminum load of 2.64 g. Respectively, all experiments were performed with 2.64 g of powder loaded, while both the powder composition and the environment composition were varied. Before the powder was introduced into the vessel, the vessel was evacuated to less than 1 torr. It was then filled with the 171 torr of O₂. The powder was introduced with a blast of a gas mixture typically comprising nitrogen and methane. The blast was produced by opening a solenoid valve connecting the vessel with a 2-gallon gas reservoir filled with nitrogen/methane gas mixture at 4,200 torr. Before each experiment, the gas mixture was prepared by evacuating the gas reservoir and re-filling it with 81.3 to 243.8 torr of methane and the balance of nitrogen. The duration of the gas blast pulse was 200 ms. At the end of the blast, the pressure in the vessel was close to 1 atm. To reduce the turbulence in produced gas powder mixture, the gas blast was followed by a 300 ms waiting period. Finally, the powder was ignited using an electrically heated tungsten wire placed in the center of the vessel. The combustion pressure traces were measured in real time using an American Sensor Technology AST 4700 transducer. The values and the rates of pressure rise produced by the combustion were compared for different powders. Condensed combustion products were collected after each run for subsequent assessment of their compositions using x-ray diffraction (XRD) analysis.

In one set of experiments, the oxidizing environment was fixed to nominally include 3% CH₄, 22% O₂, and 75% N₂. The amounts of the nanocomposite powders added to aluminum were 10, 20, and 30 mass %. The gaseous combustion products included moisture and carbon oxides, imitating the environment in actual propellants better than the previous CVE experiments performed in air [15, 16]. The second set of experiments was carried out using a constant mass % of additive for each modifier in the aluminum powder load. The methane concentration varied between 1.5 and 4.5 % while the oxygen concentration remained constant.

4 Thermodynamic equilibrium calculations

Thermodynamic equilibrium calculations were carried out for pure aluminum and aluminum with different amounts of the nanocomposite additives. The calculations were performed using the NASA chemical equilibrium and applications (CEA) code. A constant volume combustion mode was used. The atmosphere was set at a pressure of 1 atm and composition of 3% CH₄, 22% O₂, and 75% N₂. Amounts of the modifiers used were 10, 20, and 30 mass %. Figure 3 shows the maximum pressures and temperatures from the CEA calculations. Pure aluminum exhibits the highest temperature and pressure. Both temperature and pressure decrease as more modifier material is added, but the decrease is relatively small. Based on the calculations, the 8Al+MoO₃ nanocomposite additive is expected to result in a slightly higher flame temperature than any other additive considered. The differences in the predicted temperatures and pressures for different additives taken at the same mass % are very minor.

The effect of gas composition was also considered using the CEA calculations. The pressure and combustion temperatures of the powders in the constant volume combustion were calculated for the amount of modifier fixed at 20 mass %. Combustion atmospheres with 1.5, 3.0, and 4.5 % CH₄, constant 22 % O₂ and balance of N₂ were used. Figure 4 shows the calculated values for the flame pressure and temperature. Pure aluminum flames are characterized by both higher temperature and pressure. However, the differences in the predicted pressures and temperatures for pure Al and for Al with modifiers are quite small. An increase in the combustion pressure is anticipated with increase in the methane concentration for all fuels. Conversely, the flame temperatures decrease with the increasing methane concentration indicating a substantial change in the predicted make-up of the equilibrium combustion products. The predicted pressures and temperatures do not differ much among different modifiers. Both thermite compositions appear to result in slightly higher pressures at greater concentrations of methane compared to the boron-titanium composition, which is predicted to work a bit better for dry environments.

Finally, the equilibrium calculations were used to predict the compositional make-up of the condensed product anticipated for different burn rate modifiers. Two separate calculations were performed. In the first calculation, products formed at the adiabatic flame temperature were determined. In the second calculation, main species present in the adiabatic combustion products were considered as reagent species initially at the adiabatic flame temperature and pressure. They were equilibrated to room temperature (300 K). This calculation predicted the final pressure in the combustion vessel and the mole fraction of the condensed products present. Figure 5 shows the result of these calculations. Consistently with the previous calculation results, the lowest final pressures, and thus the most substantial consumption of the gaseous oxidizers is predicted for the pure Al flames. The increase in the initial concentration methane, which is an additional fuel, generally results in a decrease in the final pressure. This effect is diminished for pure Al at higher methane concentrations. The main condensed species predicted to be present in the combustion products are listed in Table 1.

5 Results and Discussion

Table 2 shows the measured values of the minimum ignition energy (MIE) for each material ignited by an electric spark. According to the standard testing methodology, ignition is registered if individual burning particles are observed. Such particles may or may not be accompanied by a self-sustaining flame. It is noted in Table 2 whether the spark ignition resulted in a self-sustaining flame, which clearly indicated more sensitive powders. The data indicate that mixing the nanocomposite powders with aluminum results in a powder that is more sensitive than the pure aluminum but substantially less sensitive than the nanocomposite material by itself. In particular, it is worth noting that the flame did not propagate in the powder mixtures, unlike in the individual nanocomposite powders.

Shown in Fig. 6 are two characteristic pressure traces from the CVE experiment. The measured maximum combustion pressures are substantially lower than those predicted by equilibrium calculations (cf. Figs. 3, 4). This difference is likely caused by both radiation heat losses and incomplete combustion of the solid fuel. The aluminum powder with the burn rate modifier additive shows both increased maximum pressure and the rate of pressure rise. The accelerated burn rate was indeed observed for all experiments using nanocomposite powders as

burn rate modifiers. However, the maximum measured pressure could be both higher and lower than that measured for pure Al powder.

Initial CVE experiments were carried out to determine the effect of various amounts of modifier as was done in the CEA calculations shown in Fig. 3. While the targeted initial pressure in the vessel before ignition was always 1 atm, the actual initial pressures varied in the range of .94 – 1.06 atm. To minimize the effect of this initial pressure variation, the results are consistently shown in terms of the measured explosion pressures divided by the measured initial pressures. Figure 7 shows respective ratios for the maximum pressures observed in explosions over corresponding values of the initial gas pressures in the vessel for different modifiers at different additive mass percents. Results for the maximum rates of pressure rise, $(dP/dt)_{max}$, are shown in Figure 8. The error bars here and below represent the standard deviations among the experimental data points. The maximum rate of pressure rise is generally proportional to the flame speed and serves as an indicator of the burn rate. Dashed lines in both Figs. 7 and 8 indicate respectively the measured values of pressure and the rate of pressure rise for the pure aluminum powder. Unlike the calculated pressures, the experimental pressures for the powders with burn rate modifiers are mostly as high as or higher than that for pure aluminum. Interestingly, the modifier predicted to result in a higher flame temperature, nanocomposite 8Al+MoO₃, results in experimental explosion pressures exceeding those measured for pure aluminum when the additive concentration is less than 30%. The maximum rates of pressure rise shown in Figure 8 are all higher for aluminum powders mixed with additives than for the pure aluminum. The most significant improvements in the burn rate are observed for 8Al+MoO₃ and 2B+Ti nanocomposite additives at 20 wt %. The effect is consistently small for 8Al+3CuO.

The effect of methane concentration was studied experimentally for the amount of the burn rate modifier fixed at 20 wt%, similar to the calculations presented in Fig. 4. The results for both the maximum pressures and rates of pressure rise are shown in Fig. 9. The changes in the explosion pressures for the pure aluminum and aluminum with 8Al+3CuO nanocomposite additive are small and the observed trends are similar to those predicted by the equilibrium calculations. Addition of the nanocomposite 8Al+MoO₃ powder results in the explosion pressures exceeding those of the pure Al for all gas compositions. The maximum explosion pressures for aluminum with nanocomposite 2B+Ti additive consistently decrease with an increase in the methane concentration, which is opposite to the trend predicted by the equilibrium calculations (cf. Fig. 4). For the latter fuel, the maximum explosion pressures are higher than those for pure Al at lower methane concentrations. For all fuels, the rate of combustion proportional to the measured values of $(dP/dt)_{max}$ increase at the increasing methane concentrations. The most substantial improvement over pure aluminum is observed for the fuel with nanocomposite 8Al+MoO₃ powder. The increase in the burn rate is also substantial for the aluminum mixed with nanocomposite 2B+Ti.

The results presented in Fig. 9 can also be considered in terms of combustion efficiency described based on the direct comparison of the experimental and predicted explosion pressures. Specifically, ratios of the experimental maximum pressures to the respective pressures calculated by CEA code for the same initial conditions (cf. Fig. 4) are shown in Fig. 10. The trends observed in Fig. 10 are very similar to those in Fig. 9.

The final pressures in the vessel measured after the combustion was completed and the vessel was cooled down to room temperature are indicative of the oxygen consumption. These

pressures were compared to the predicted final pressures shown in Fig. 5. The ratios of the measured over calculated final pressures for different experimental conditions are shown in Fig. 11. Generally, the higher are the ratios shown in Fig. 11, the less efficient was the combustion in terms of consumption of gaseous oxidizer. For pure Al, the ratios are always greater than 1 suggesting that, as expected, combustion was less efficient than predicted by equilibrium calculation. It is interesting that for pure Al the pressure ratio shown in Fig. 11 increases with increased CH₄ concentration. No such trend is observed for aluminum powders burning with nanocomposite additives. It is also interesting that the pressure ratios are in many cases less than 1 (the level of pressure ratio equal to 1 is highlighted by a dashed line). Combustion cannot, of course, be more efficient than predicted by the equilibrium calculations, but it is very possible that the calculations did not take into account complex oxides than can form in presence of multiple fuels. Production of additional condensed fully or partially oxidized compounds can substantially increase the consumption of the gaseous oxygen and thus improve the burning efficiency. In addition, formation of oxy-nitrides and oxy-carbides is possible in the combustion systems considered here. Such complex compounds are difficult to identify from bulk sample XRD analysis described below and a more detailed study of the combustion products would be desired in the future in order to interpret the observed trend in more detail. The trends observed in Fig. 11 can also be qualitatively correlated to the ratio of the experimental to calculated maximum pressures. The highest maximum pressure attained in the flame corresponds to the lowest final pressure measured after the combustion products are cooled. A simplistic reasoning is that attaining higher pressure means more consumption of gaseous oxidizer in turn resulting in a lower final pressure. This correlation works reasonably well for pure Al and thermite type additives, while the trend is less clear for the 2B+Ti nanocomposite additive. This is likely because the latter additive affects the overall product composition most substantially, chiefly due to a relatively low boiling point of boron oxide.

The combustion products from the explosion vessel were collected and analyzed by XRD. Table 1 lists the products identified by XRD and the ones predicted from the CEA calculations. The most significant discrepancies are detected for the fuel system using the nanocomposite 8Al+3CuO additive. In addition to the partially oxidized copper, Cu₂O not predicted by CEA, substantial presence of unoxidized Cu-Al alloy, CuAl₂ was detected by XRD. It is likely that formation of this alloy resistive to oxidation resulted in a relatively low combustion pressures observed in respective experiments. Note that although some of the predicted products were not identified by XRD, they could still be present in the combustion products and not detected because of a relatively low sensitivity of the bulk XRD analysis.

6 Conclusions

Addition of relatively small amounts of nanocomposite burn rate modifiers to aluminum enables substantial increase in its burn rate in gaseous oxidizers without a significant reduction in the overall theoretical combustion enthalpy. The improvements in the burn rates are observed experimentally in the oxygenated environments including products of hydrocarbon combustion. Most importantly, additives of nanocomposite powders of 8Al+MoO₃ and 2B+Ti to micron-sized aluminum were found to be effective in increasing both the rate of pressure rise and maximum pressure in the respective constant volume explosion experiments. It was observed that 20 wt % of additive resulted in the best combination of the achieved burn rate and pressure. Additives of

nanocomposite 8Al+3CuO powder did not show appreciable improvements in aluminum combustion, most likely due to formation of an oxidation resistant CuAl₂ alloy.

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Table 1. Condensed combustion products predicted by CEA calculation and identified by XRD

Species	Calculated	Experimental (XRD)
Al		X
Al ₂ O ₃	X	X
Al(OH) ₃	X	
Cu	X	
CuO	X	X
Cu ₂ O		X
CuAl ₂		X
MoO ₃	X	X
MoO ₂	X	
B ₂ O ₃	X	X
HBO ₂	X	
TiO ₂	X	X
C (graphite)	X	

Table 2. Ignition sensitivity of materials to electro-static discharge

Material	MIE (mJ)	Self-sustaining flame
Spherical Al 10-14 micron	25.7	No
8Al+3CuO nanocomposite	3.8	Yes
8Al+MoO ₃ nanocomposite	<0.8	Yes
2B+Ti nanocomposite	1.2	No
Al (10-14 micron) + 20%(8Al+3CuO) blend	13.2	No
Al (10-14 micron) + 20%(8Al+MoO ₃) blend	6.9	No
Al (10-14 micron) + 20%(2B+Ti) blend	1.9	No

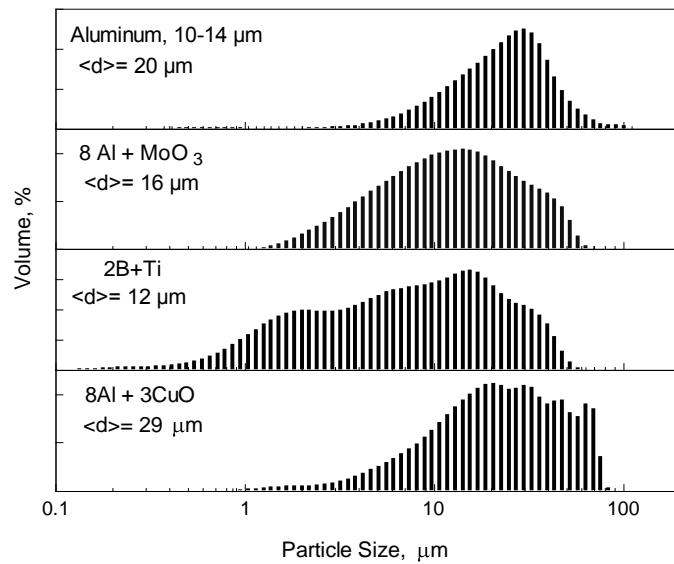


Fig. 1. Particle size distributions for the different powders used in this project. Volume mean particle sizes are shown for each materials.

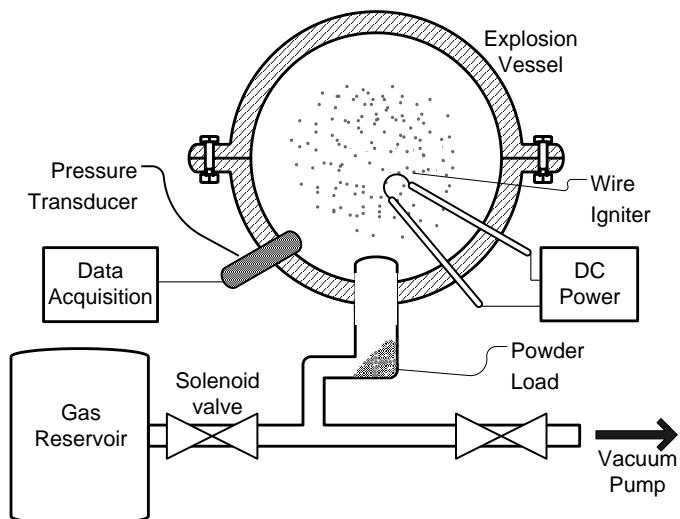


Fig. 2. Schematic diagram of the constant volume explosion apparatus

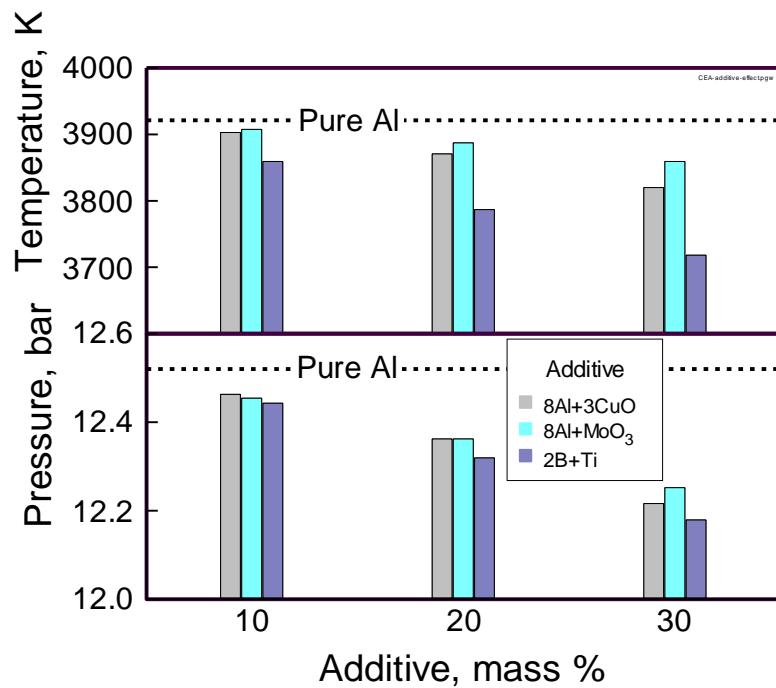


Fig. 3. Adiabatic flame temperatures and respective pressures predicted by CEA calculations for constant volume combustion of different solid fuels in a fixed environment: 3%CH₄, 22% O₂; 75% N₂, initially at 1 atm. Solid fuels are aluminum with different mass % of burn rate additives.

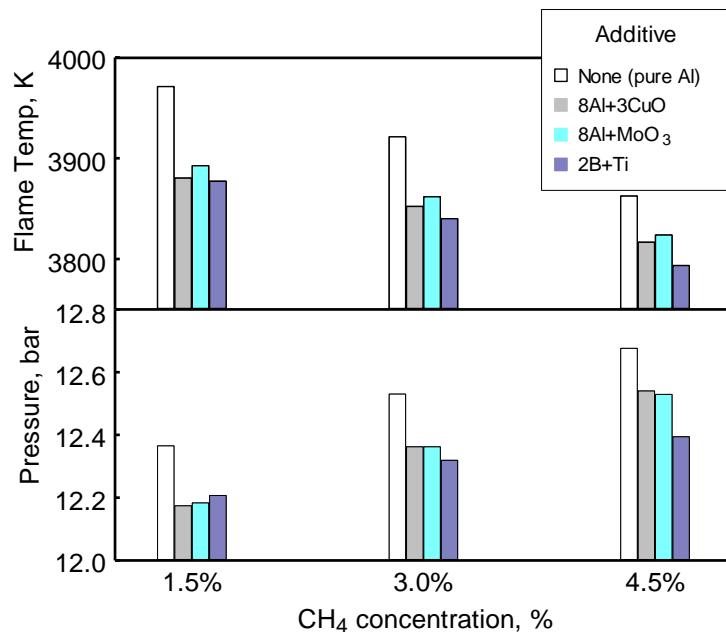


Fig. 4. Adiabatic flame temperatures and respective pressures predicted by CEA calculations for constant volume combustion of aluminum with 20 wt % of different additives in different gas environments. For each case, 75% N₂ is balanced by O₂ and CH₄ initially at 1 atm.

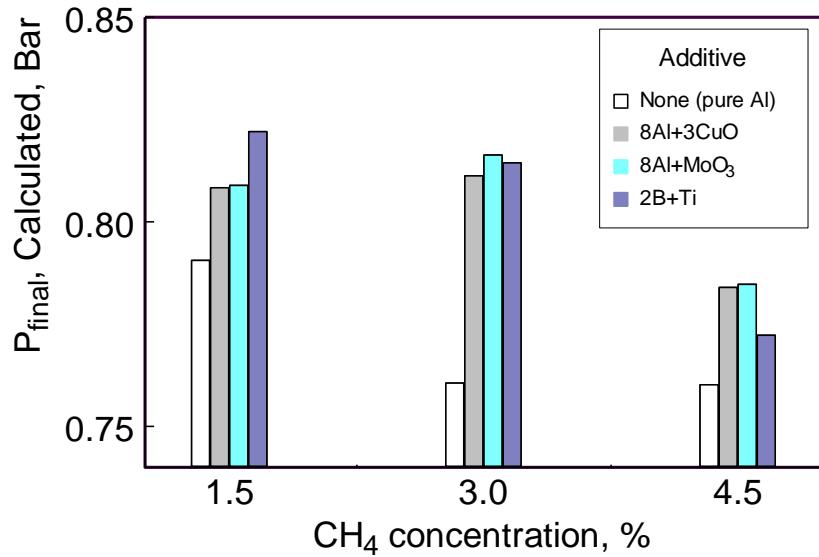


Fig. 5. Final pressure in the explosion vessel after combustion is completed predicted by a CEA calculation using the adiabatic pressure, temperature, and combustion products as the input and preset to equilibrate to room temperature. For each case, 75% N₂ is balanced by O₂ and CH₄ initially at 1 atm.

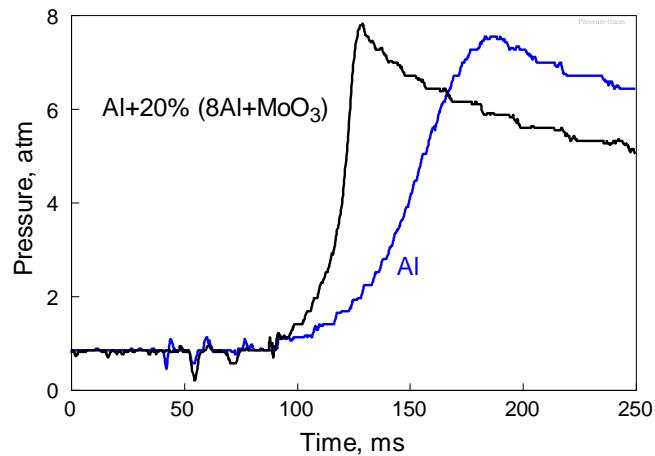


Fig. 6. Pressure traces from constant volume explosion experiments for pure aluminum powder and aluminum powder with 20 wt % of a nanocomposite additive. The gas environment is 3% CH₄, 22% O₂, and 75% N₂.

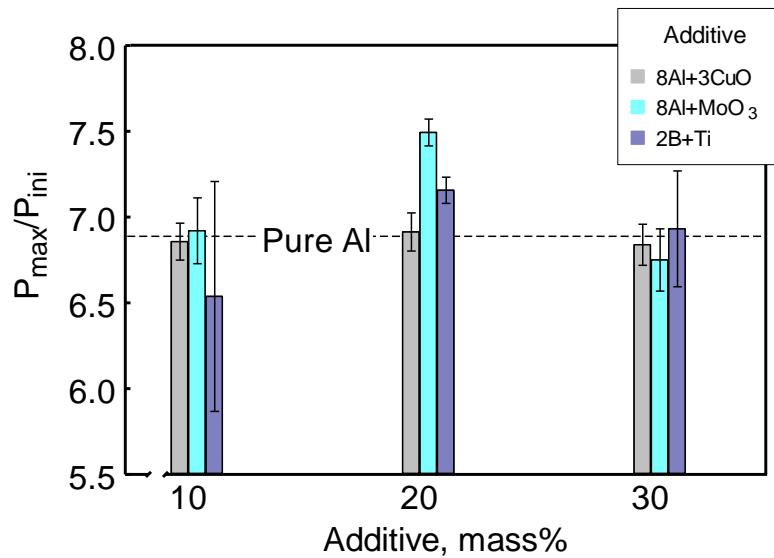


Fig. 7. Normalized maximum combustion pressure measured in CVE experiments for aluminum powders with varied amounts of fuel additives. The gas environment is fixed at 3% CH₄, 22% O₂, and 75% N₂.

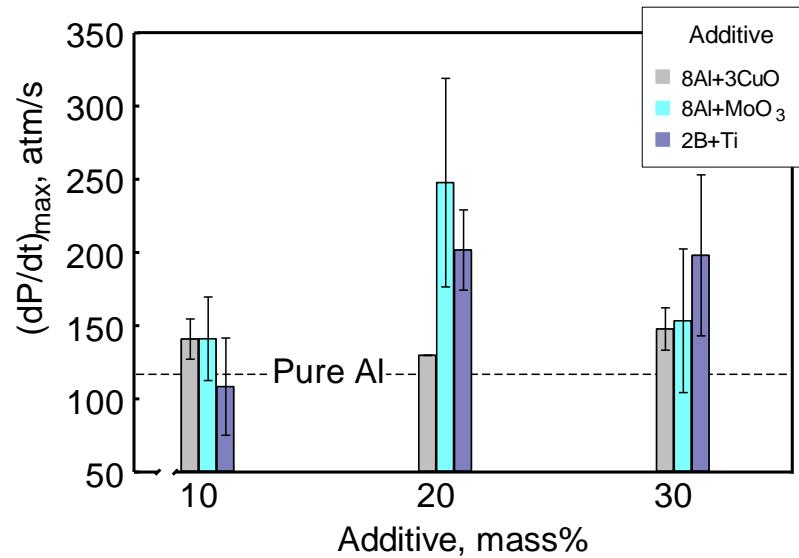


Fig. 8. Maximum rates of pressure rise measured in CVE experiments for aluminum powders with varied amounts of fuel additives. The gas environment is fixed at 3% CH₄, 22% O₂, and 75% N₂.

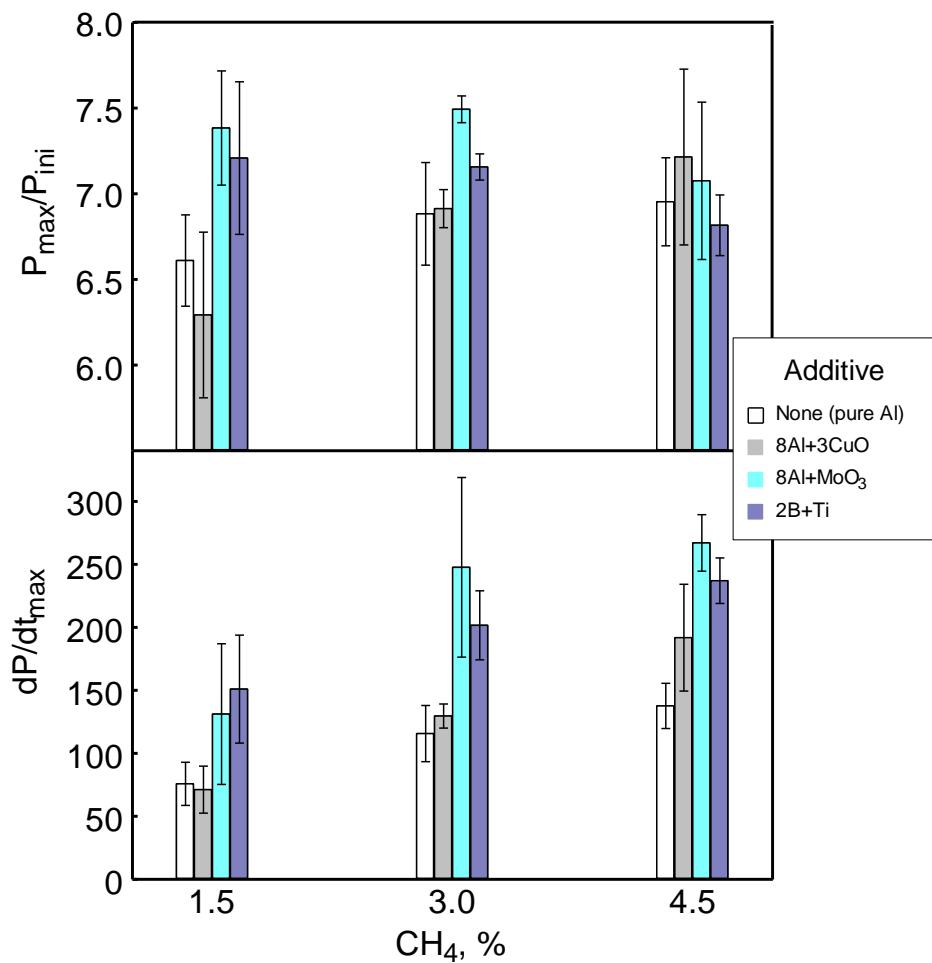


Fig. 9. Normalized maximum combustion pressures and maximum rates of pressure rise measured in CVE experiments for aluminum powders with 20 wt % of different fuel additives. The gas environments are varied with 1.5, 3, and 4.5 % of CH₄, 22% O₂, and balance of N₂.

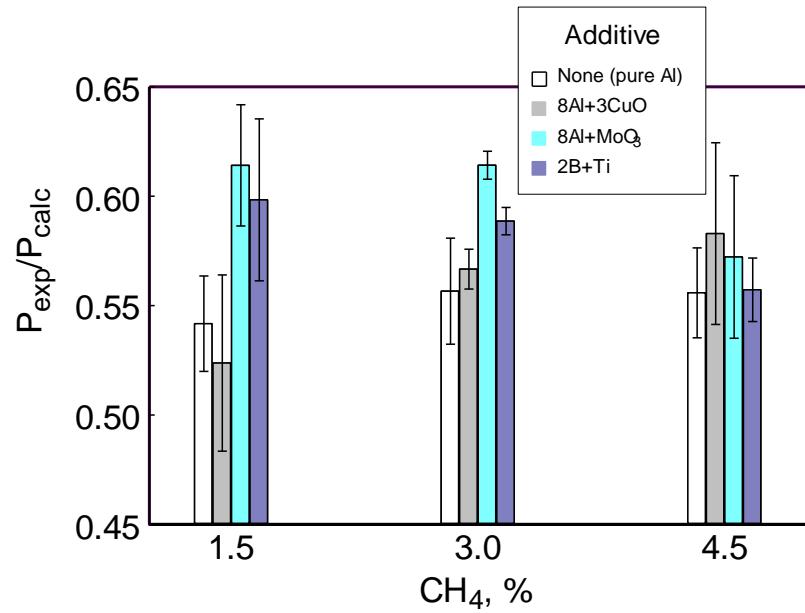


Fig. 10. Ratios of the experimental maximum pressures to the respective pressures calculated by CEA for the same initial conditions (cf. Fig. 4, varied oxidizing environments).

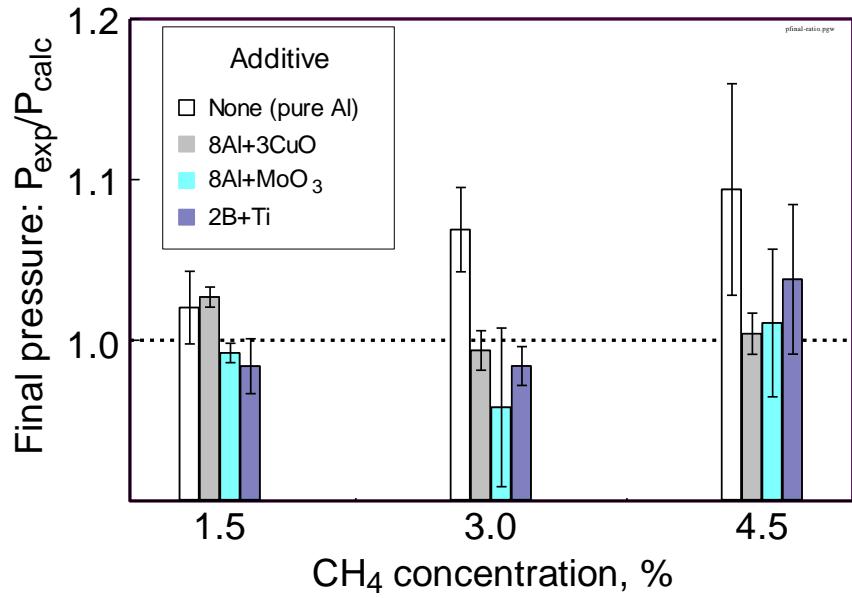


Fig. 11. Ratios of the measured over calculated final pressures in the explosion vessel for the combustion products cooled to room temperature for varied initial oxidizing environments.